

6.0 ANALYTICAL PROCEDURES

6.1 LABORATORY PROCEDURES

The laboratory procedures to be performed include methodologies from the United States Environmental Protection Agency (USEPA), Washington State Department of Ecology Analytical Methods for Petroleum Hydrocarbons, American Society for Testing and Materials (ASTM) Annual Book of ASTM Standards, and the Puget Sound Water Quality Authority (PSWQA, 1996) as presented in Table 6-1. All samples will be analyzed following the USACE Shell guidance present in Appendix I of EM 200-1-3 (USACE, 2001). Briefly described below are the sample preparation and analytical methods to be performed.

6.1.1 Semivolatile Organic Compounds (SVOCs)

6.1.1.1 Sample Preparation

Sediment sample preparation required prior to analysis for SVOCs will be performed in accordance with USEPA Method 3550B, as referenced in Table 6-1. Sample cleanup may be performed by Gel Permeation Chromatography (GPC) by USEPA Method 3640A if necessary. Additional cleanups (i.e., USEPA Method 3650B acid-base partition cleanup, or USEPA Method 3660B sulfur cleanup) may be applied if necessary.

6.1.1.2 Analytical Method

The extract will be analyzed for SVOCs by GC/MS in accordance with USEPA Method 8270C (Table 6-1). The reporting limits (RLs) for SVOCs are presented in Table 6-2. The RLs are on a wet-weight basis. Sample RLs are highly matrix-dependent. RLs are provided as guidance and may not always be achievable.

6.1.2 Polychlorinated Biphenyls (PCBs) - Aroclors

6.1.2.1 Sample Preparation

Sediment sample preparation required prior to analysis for PCBs will be performed in accordance with USEPA Method 3550B, as referenced in Table 6-1. Sample cleanup will be performed by sulfur by USEPA Method 3660B. Additional cleanups (i.e., USEPA Method 3620B florisil cleanup, USEPA Method 3630C silica gel cleanup, or USEPA Method 3665A sulfuric acid/permanganate cleanup) may be applied if necessary.

6.1.2.2 Analytical Method

The extract will be analyzed for PCBs (Aroclors) by gas chromatography (GC) utilizing an electron capture detector (ECD) in accordance with USEPA Method 8082 (Table 6-1). The RLs

for PCB Aroclors are presented in Table 6-2. The RLs are on a wet-weight basis. Sample RLs are highly matrix-dependent. RLs are provided as guidance and may not always be achievable.

6.1.3 Organochlorine Pesticides (Pesticides)

6.1.3.1 Sample Preparation

Sediment sample preparation required prior to analysis for pesticides will be performed in accordance with USEPA Method 3550B, as referenced in Table 6-1. Sample cleanup will be performed by florisil by USEPA Method 3620B. Additional cleanups (i.e., USEPA Method 3640B GPC cleanup) may be applied if necessary.

6.1.3.2 Analytical Method

The extract will be analyzed for pesticides by GC/ECD in accordance with USEPA Method SW8081A (Table 6-1). The RLs for pesticides are presented in Table 6-2. The RLs are on a wet-weight basis. Sample RLs are highly matrix-dependent. RLs are provided as guidance and may not always be achievable.

6.1.4 Diesel and Heavy Oil Range Organics

6.1.4.1 Sample Preparation

Sediment sample preparation prior to analysis for diesel and heavy oil range organics will be performed in accordance with USEPA Method 3550B, as referenced in Table 6-1. Sample cleanup by USEPA Method 3620B florisil cleanup or USEPA Method 3630C silica gel cleanup may be applied if necessary.

6.1.4.2 Analytical Method

Diesel and heavy oil range organics analysis will be performed following Washington State Department of Ecology Method Northwest Total Petroleum Hydrocarbon – Diesel Range Organics (NWTPH-Dx) for solid matrices (WDOE, 1997). The RL for diesel range hydrocarbons is presented in Table 6-2. The RLs are on a wet-weight basis. Sample RLs are highly matrix-dependent. RLs are provided as guidance and may not always be achievable.

6.1.5 Butyltins

6.1.5.1 Sample Preparation

Sediment samples for butyltins are prepared by methylene chloride/tropolone extraction and be silica/alumina cleanup.

6.1.5.2 Analytical Method

Sample digestates will be analyzed by GC/MS as described in the Krone, 1988 method reference in Table 6-1.

6.1.6 Total Metals (except mercury)**6.1.6.1 Sample Preparation**

Sediment sample preparation for total metals involves acid digestion prior to analysis. This will be performed in accordance with USEPA Method 3050B (Acid Digestion of Sediments, Sludges and Soils), as referenced in Table 6-1.

6.1.6.2 Analytical Method

Total metal digestates will be analyzed following USEPA Method 6020, as presented in Table 6-1. The RLs for total metals are presented in Table 6-2. The RLs are on a wet-weight basis. Sample RLs are highly matrix-dependent. RLs are provided as guidance and may not always be achievable.

6.1.7 Mercury**6.1.7.1 Sample Preparation**

Sediment sample preparation for mercury involves digestion prior to analysis, as described in USEPA Method 7471A.

6.1.7.2 Analytical Method

Mercury will be analyzed following USEPA Method 7471A for solid matrices. The RL for mercury is presented in Table 6-2. The reporting limit is on a wet-weight basis. Sample RLs are highly matrix-dependent. RLs are provided as guidance and may not always be achievable.

6.1.8 Total Organic Carbon (TOC)**6.1.8.1 Sample Preparation**

Sediment sample preparation will be performed in accordance with Method 9060, as referenced in Table 6-1.

6.1.8.2 Analytical Method

Total organic carbon (TOC) will be analyzed following USEPA Method 9060. The RL for TOC is presented in Table 6-2. The reporting limit is on a wet-weight basis. Sample RLs are highly matrix-dependent. RLs are provided as guidance and may not always be achievable.

6.1.9 Grain Size**6.1.9.1 Sample Preparation**

Sediment samples do not require preparation prior to analysis, as referenced in Table 6-1, but may require cleanup for anticoagulation.

6.1.9.2 Analytical Method

Grain size will be determined following ASTM Method D422.

6.1.10 pH

Sediment pH will be measured in accordance with Method SW9045C, as referenced in Table 6-1. Water samples will be measured for pH in accordance with Method 150.1, as referenced in Table 6-1.

6.1.11 Moisture Content

Moisture content will be determined following ASTM Method D2216-90.

6.1.12 Acid Volatile Sulfides (AVS) and Simultaneously Extracted Metals (SEM)**6.1.12.1 Sample Preparation**

The sediment sample is prepared for AVS and SEM with hydrochloric acid as specified in the EPA Draft Method as referenced in Table 6-1. The acidified sediment sample is membrane filtered before SEM determination.

6.1.12.2 Analytical Method

Hydrogen sulfide will be determined by the colorimetric method as described in the EPA Draft Method referenced in Table 6-1. SEM will be analyzed by EPA Methods 6020 and 7471 (see Section 6.1.6 and 6.1.7).

6.1.13 Total Suspended Solids (TSS)

Water samples will be measured for TSS in accordance with Method 160.2, as referenced in Table 6-1.

6.1.14 Dissolved Organic Carbon (DOC)

Water samples will be filtered in the field and measured for DOC in accordance with Method 415.1, as referenced in Table 6-1.

6.1.15 Hardness

Water samples will be measured for hardness as calcium carbonate in accordance with Method 130.2, as referenced in Table 6-1.